AI-66-87

INVESTIGATION OF SUPERCURRENT INSTABILITIES IN TYPE II SUPERCONDUCTORS

GPO PRICE $

CFSTI PRICE(S) $

Hard copy (HC) 1.00

Microfiche (MF) .50

ff 653 July 65

FIRST QUARTERLY PROGRESS REPORT

CONTRACT NAS8-20552
MARSHALL SPACE FLIGHT CENTER
HUNTSVILLE, ALABAMA

N66 29908
(ACCESSION NUMBER)

(THRU)

21
(PAGES)

1
(CODE)

CR-76060
(NASA CR OR TMX OR AD NUMBER)

26
(CATEGORY)

FACILITY FORM 602

ATOMICS INTERNATIONAL

A DIVISION OF NORTH AMERICAN AVIATION, INC.

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MAY 13, 1966

CONTENTS

FIGURES

I. INTRODUCTION

The summary report for the predecessor contract* shows how the current density and other aspects of superconductivity are influenced by heat treatment and plastic deformation in Ti-22 at. % Nb. It is therefore necessary to examine quantitatively and in detail the microstructure of this alloy after various treatments to correlate it with superconducting behavior.

Specific microstructural features as precipitates or closely spaced internal boundaries that are likely to be important are not resolvable with the light microscope but may be revealed with the electron microscope. Light microscopy is necessary to detect large-scale phase changes as well as grain size and possible inhomogeneities.

The β phase (body-centered cubic) in Ti-22 at. % Nb may transform from a state of metastable equilibrium by precipitating other metastable phases prior to the appearance of the equilibrium α phase. Plastic deformation and annealing, separately or in combination, brings on decomposition of the β phase. The task ahead is to identify the reaction products and measure the density of potential flux-pinning features of the microstructure.

*NAS8-5356

II. TRANSFORMATIONS IN THE ANNEALED β PHASE

Samples of Ti-22 at. % Nb were annealed 3 hours at 800°C and rapidly cooled to retain the β phase. They were subsequently re-annealed in the temperature range 400 to 550°C — in the two-phase $\alpha + \beta$ region on the equilibrium diagram — to initiate transformation. Figures 1 to 5 are light photomicrographs showing samples before and after the transformation anneal. No precipitation is detected after 1 hour at 400°C. After 24 hours at 450°C, particles appear in the matrix and on grain boundaries. The sample annealed 2 hours at 500°C contains what is probably equilibrium α phase that has nucleated at grain boundaries. A much smaller amount of this is seen in the sample annealed 1 hour at 550°C.

Electron microscope samples were prepared from bulk samples by chemically or electrolytically thinning. Electron diffraction patterns are obtained for each photomicrograph to orient the foil and to solve the crystal structure of the precipitating phase. The former task is relatively simple while the latter is frequently difficult.

Figures 6 to 9 reveal precipitation in samples annealed 400, 450, and 500°C. After 1 hour at 400°C precipitation is in an early stage. Contrast between particles and matrix is not strong owing to the fact that the crystal structure of the particle is not far removed from that of the matrix. The average particle size is less than 500 Å and the particle density is on the order of 10^{15} cm^{-3}. Electron diffraction gave negative results.

Annealing 24 hours at 450°C has yielded plate-shaped precipitates having strong contrast. Electron diffraction data has shown that the plates lie parallel to (111) matrix planes and that the precipitate is the ω(omega) phase, common in group IV-V binary alloys. The density of the particles is still about 10^{15} cm^{-3} and the large dimension of the plate is around 1500 Å.

The two electron photomicrographs of samples transformed at 500°C show the growth of particles from 15 minutes to 2 hours. Electron diffraction patterns have been obtained but have proven unsolvable to date. The density of particles after 15 minutes is about 10^{14} cm^{-3} and the size is about 2000 Å.

III. TRANSFORMATIONS IN PLASTICALLY DEFORMED β PHASE

Alloy samples were rolled to a 2:1 reduction after rapidly cooling from an 800°C anneal. The deformation caused the metastable β phase to transform as seen in Figure 10. It is likely that part of the volume remains untransformed as a 2:1 reduction is not a severe deformation. Subsequent annealing in the range 350 to 500°C produces further transformation as shown in Figures 11 to 14. Light microscopy offers little insight into the complexities of this process.

IV. FUTURE WORK

1. Critical current vs magnetic field tests will commence on samples whose structure is known from electron microscopy. Quantitative statistical particle measurements will be made on these samples.
2. Electron microscopy will continue on samples transformed from the annealed state and will start on samples transformed in the plastically deformed state.
3. X-ray diffraction will be used to identify the transformation product found after rolling.

V. ACKNOWLEDGMENT

The light and electron microscopy was performed by C. G. Rhodes and R. A. Spurling at the North American Aviation Science Center.

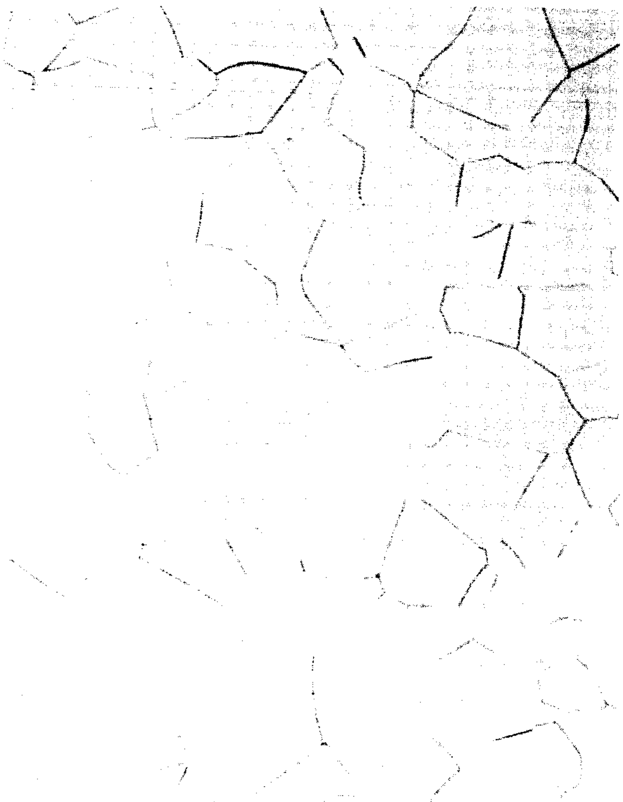

Figure 1. Ti-22 at. % Nb Showing Retained β Phase. (250X)

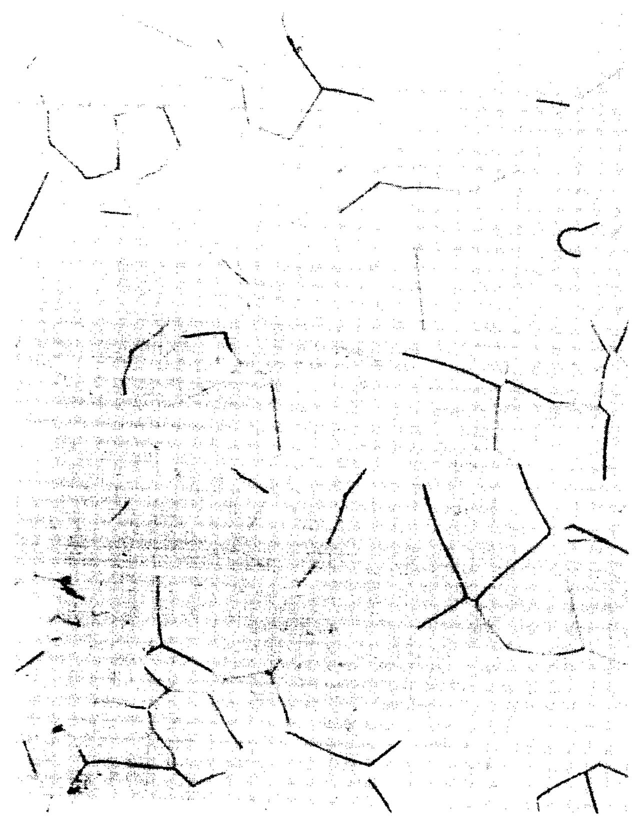

Figure 2. Ti-22 at. % Nb Annealed 1 Hour at 400°C. No transformation product is visible. (250X)

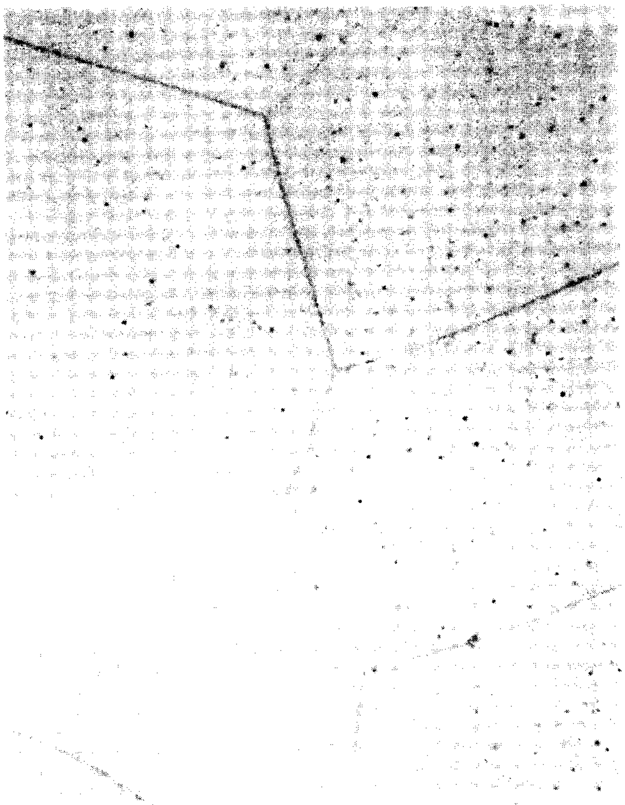

Figure 3. Ti-22 at. % Nb Annealed 24 Hours at 450°C. Precipitation is seen at grain boundaries and in grains. (1000X)

Figure 4. Ti-22 at. % Nb Annealed 2 Hours at 500°C. The α phase is seen growing from grain boundaries. (250X)

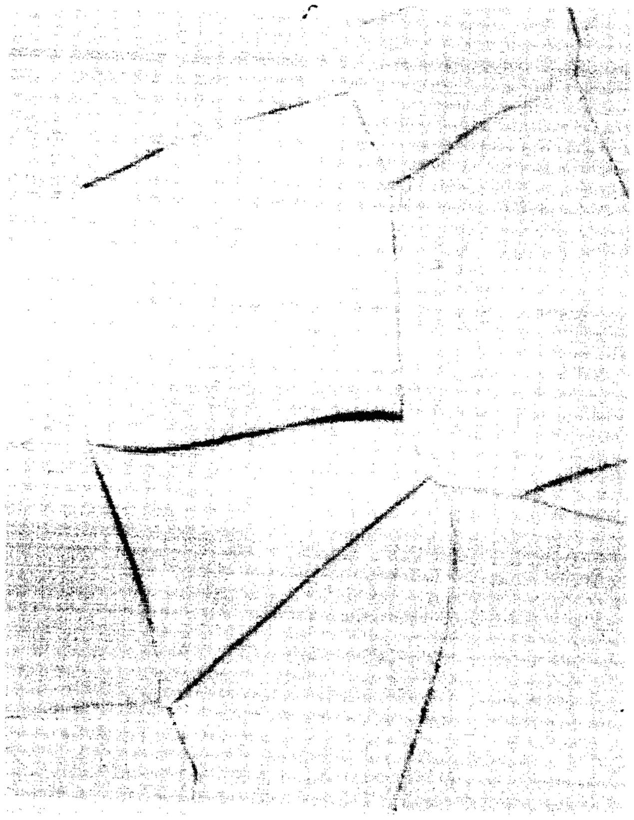

Figure 5. Ti-22 at. % Nb Annealed 1 Hour at 550°C. Some precipitation, probably α phase, is seen on grain boundaries.

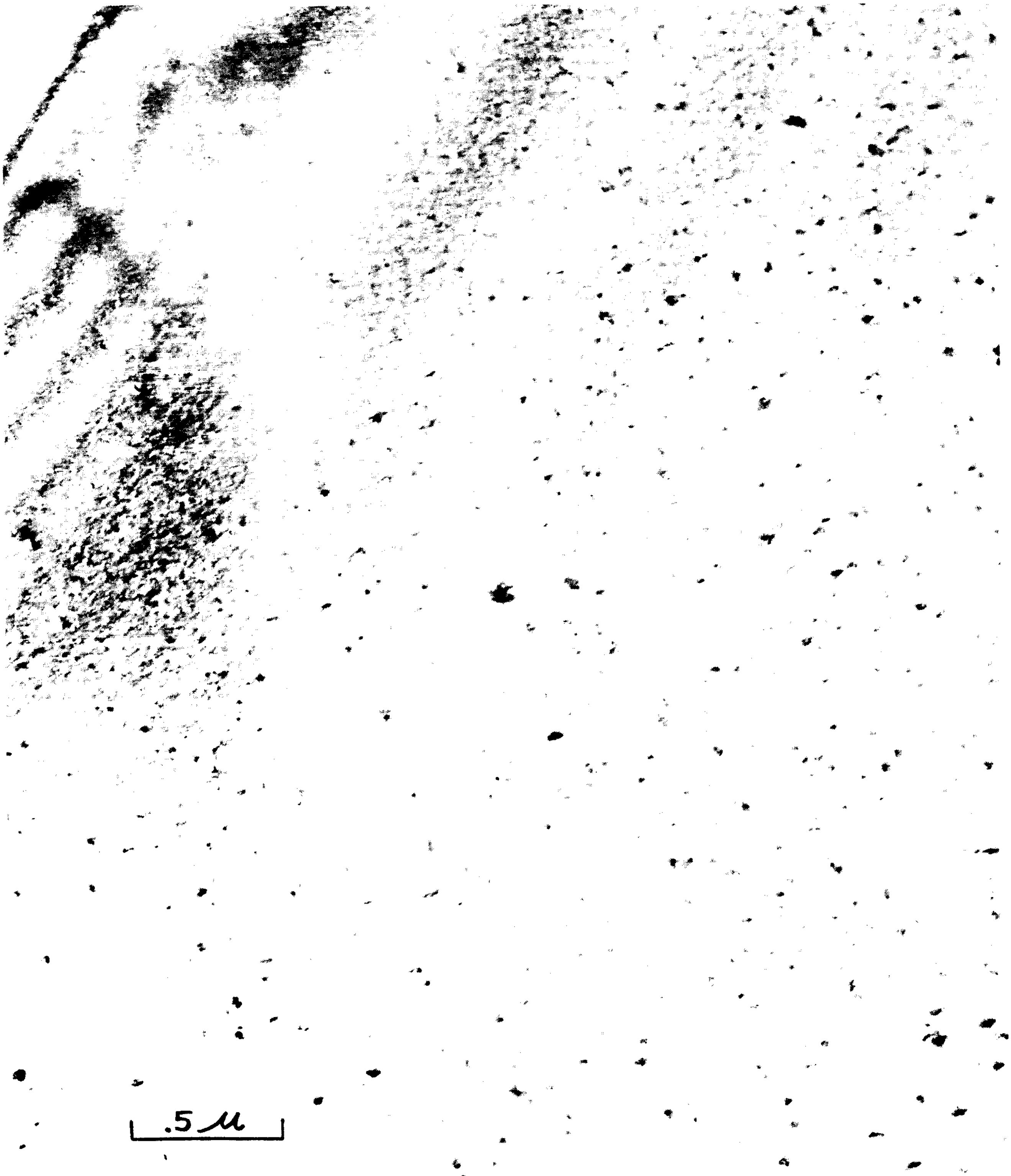

Figure 6. Ti-22 at. % Nb Annealed 1 Hour at 400° C. Transmission electron photomicrograph showing an early stage of precipitation. Compare with Figure 2.

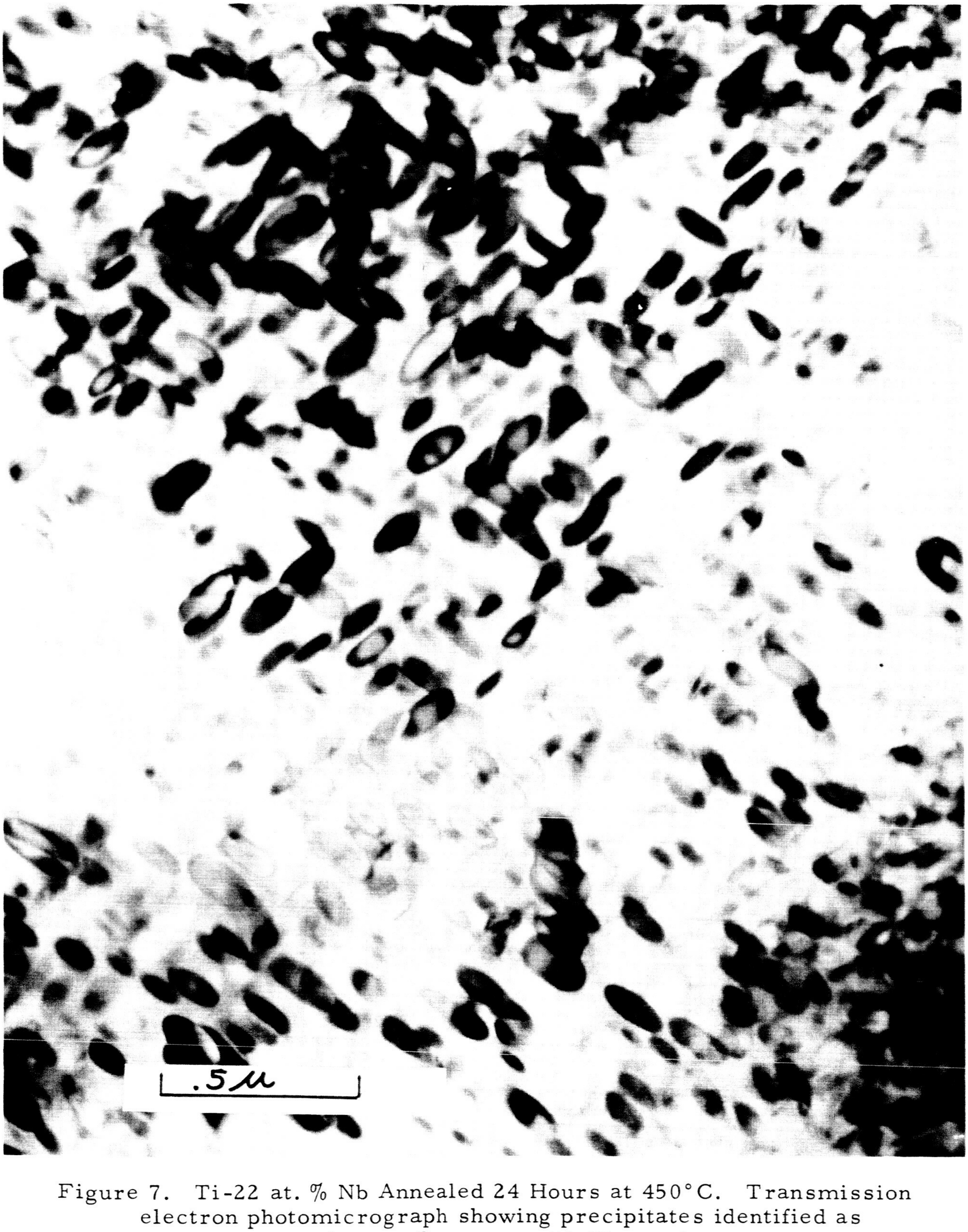

Figure 7. Ti-22 at. % Nb Annealed 24 Hours at 450°C. Transmission electron photomicrograph showing precipitates identified as ω phase. Compare with Figure 3.

Figure 8. Ti-22 at. % Nb Annealed 15 Minutes at 500°C. Transmission electron photomicrograph showing unidentified precipitates.

Figure 9. Ti-22 at. % Nb Annealed 2 Hours at 500°C. Transmission electron photomicrograph showing an advanced stage of precipitation.

Figure 10. Ti-22 at. % Nb Reduced 2:1 by Rolling Showing the Resulting Transformation. (1000X)

Figure 11. Ti-22 at. % Nb Reduced 2:1 by Rolling and Annealed 1 Hour at 350°C. (1000X)

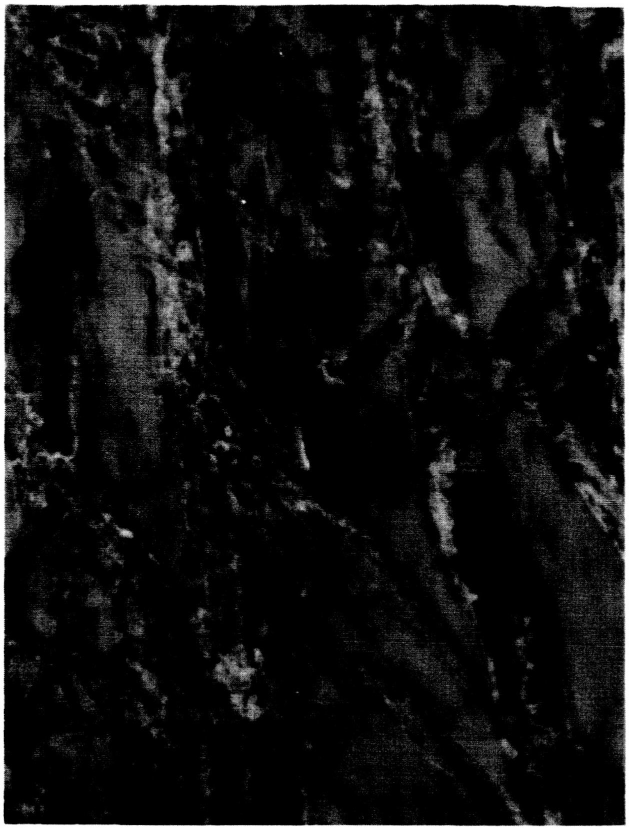

Figure 12. Ti-22 at. % Nb Reduced 2:1 by Rolling and Annealed 1 Hour at 400°C.(1000X)

Figure 13. Ti-22 at. % Nb Reduced 2:1 by Rolling and Annealed 1 Hour at 450°C.(1000X)

Figure 14. Ti-22 at. % Nb Reduced 2:1 by Rolling and Annealed 1 Hour at 500°C. (1000X)